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Notice of Allowability	Application No.	Applicant(s)	
	10/009,863	SCHABRON ET AL.	
	Examiner	Art Unit	
	Yelena G. Gakh, Ph.D.	1743	

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address--

All claims being allowable, PROSECUTION ON THE MERITS IS (OR REMAINS) CLOSED in this application. If not included herewith (or previously mailed), a Notice of Allowance (PTOL-85) or other appropriate communication will be mailed in due course. **THIS NOTICE OF ALLOWABILITY IS NOT A GRANT OF PATENT RIGHTS.** This application is subject to withdrawal from issue at the initiative of the Office or upon petition by the applicant. See 37 CFR 1.313 and MPEP 1308.

1. ☒ This communication is responsive to Interviews held on 03/14-03/18/04 with Nicole Ressue and the Examiner's Amendment.
2. ☒ The allowed claim(s) is/are 94-125.
3. ☐ The drawings filed on _____ are accepted by the Examiner.
4. ☐ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).
 - a) ☐ All b) ☐ Some* c) ☐ None of the:
 1. ☐ Certified copies of the priority documents have been received.
 2. ☐ Certified copies of the priority documents have been received in Application No. _____.
 3. ☐ Copies of the certified copies of the priority documents have been received in this national stage application from the International Bureau (PCT Rule 17.2(a)).

* Certified copies not received: _____.

Applicant has THREE MONTHS FROM THE "MAILING DATE" of this communication to file a reply complying with the requirements noted below. Failure to timely comply will result in ABANDONMENT of this application.

THIS THREE-MONTH PERIOD IS NOT EXTENDABLE.

5. ☐ A SUBSTITUTE OATH OR DECLARATION must be submitted. Note the attached EXAMINER'S AMENDMENT or NOTICE OF INFORMAL PATENT APPLICATION (PTO-152) which gives reason(s) why the oath or declaration is deficient.
 6. ☒ CORRECTED DRAWINGS (as "replacement sheets") must be submitted.
 - (a) ☐ including changes required by the Notice of Draftsperson's Patent Drawing Review (PTO-948) attached
 - 1) ☐ hereto or 2) ☐ to Paper No./Mail Date _____.
 - (b) ☒ including changes required by the attached Examiner's Amendment / Comment or in the Office action of Paper No./Mail Date _____.
- Identifying indicia such as the application number (see 37 CFR 1.84(c)) should be written on the drawings in the front (not the back) of each sheet. Replacement sheet(s) should be labeled as such in the header according to 37 CFR 1.121(d).**
7. ☐ DEPOSIT OF and/or INFORMATION about the deposit of BIOLOGICAL MATERIAL must be submitted. Note the attached Examiner's comment regarding REQUIREMENT FOR THE DEPOSIT OF BIOLOGICAL MATERIAL.

Attachment(s)

- | | |
|--|--|
| 1. <input checked="" type="checkbox"/> Notice of References Cited (PTO-892) | 5. <input type="checkbox"/> Notice of Informal Patent Application (PTO-152) |
| 2. <input type="checkbox"/> Notice of Draftsperson's Patent Drawing Review (PTO-948) | 6. <input checked="" type="checkbox"/> Interview Summary (PTO-413),
Paper No./Mail Date <u>031704</u> |
| 3. <input checked="" type="checkbox"/> Information Disclosure Statements (PTO-1449 or PTO/SB/08),
Paper No./Mail Date <u>12/10/01</u> | 7. <input checked="" type="checkbox"/> Examiner's Amendment/Comment |
| 4. <input type="checkbox"/> Examiner's Comment Regarding Requirement for Deposit
of Biological Material | 8. <input checked="" type="checkbox"/> Examiner's Statement of Reasons for Allowance |
| | 9. <input type="checkbox"/> Other _____ |

DETAILED ACTION

1. Preliminary Amendment filed on 12/10/01 is acknowledged. Claims 1-16, 18, 22, 30-31, 33-36, 49, 57-59, 60-64, 79-80 and 84-93 are cancelled without prejudice. Claims 17, 19-21, 23-29, 32, 37-48, 50-57 and 60-78 are pending in the application.

Drawings

2. The amended claims are allowed (see below). Corrected drawings (Figures 8-12) should be submitted because of their poor quality. The drawings from the parent case are suggested for replacement.

EXAMINER'S AMENDMENT

3. An examiner's amendment to the record appears below. Should the changes and/or additions be unacceptable to applicant, an amendment may be filed as provided by 37 CFR 1.312. To ensure consideration of such an amendment, it **MUST** be submitted no later than the payment of the issue fee.

Authorization for this examiner's amendment was given in a telephone interview with Nicole Ressue in a series of Interviews on 03/11/04-04/18/04.

The application has been amended as follows:

Replace the abstract with the following paragraph:

VII. ABSTRACT

Indicia of stability or thresholds of instability may be used to assess the proximity of hydrocarbon materials such as petroleum residua to deposition of carbon rich materials. The invention may be used to evaluate suitability of such hydrocarbon materials to various types of processing methodologies, or to determine processing parameters, either prior to processing or during processing. Prediction of proximity to deposition of carbon rich materials may result in continuous process parameters or increased output, decreased energy use or decreased amount of

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emissions per unit of such hydrocarbon materials compared to conventional processing technology. Products produced or products processed with this technology may also have a higher level of purity.

In the specification:

On page 6, line 17, delete “to” and replace with “and”.

On page 7, lines 10-13 delete, [or the size ratio of a plurality of solvated core materials in comparison to the size of the plurality solvated of core materials with associated solvent (such as trapped solvent between them) (K_F),] and replace with "or the ratio of an average size of a plurality of solvated core materials with an associated solvent to an average size of a plurality of solvated core materials, thus representing an amount of associated solvent associated with a plurality of solvated core materials (K_F), ".

On page 7, line 27 after, “polarity.” add “Of course, the term ‘amount’ generally, may include any of different kinds of measurements such as, but not limited to, quantities, lengths, sizes, volumes, weights, weight percentages, weight fractions, volume percentages, volume fractions, radii, diameters, circumferences, or the like, however measured such as but certainly not limited to ultrasound spectroscopy, microscopy, filtration, solvation, titration, NMR spectroscopy, NMR tomography, mass spectrometry, infrared spectrometry, infrared Raman spectroscopy, size exclusion chromatography, gel electrophoresis, paper chromatography, or the like.”

On page 36, line 7, delete “to” and replace with “and”.

On page 36, line 9, after “materials” add “(with associated solvent)”.

On page 36, line 9 delete “other associated solvent”.

On page 36, line 9, add “solvated core materials” before “(K_F) when”.

On page 36, line 22, after “solvated core materials to the” add “solvated”.

In the claims:

Cancel claims 17, 19-21, 23-29, 32, 37-48, 50-57 and 60-78.

Add new claims 94-125:

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94. A method of processing petroleum residua, comprising the steps of:
- providing an amount of said petroleum residua having solvated asphaltenes dispersed in said petroleum residua;
 - precipitating an amount of said asphaltenes from said amount of said petroleum residua with a first solvent and determining said amount of precipitated asphaltenes;
 - dissolving at least part of said amount of said precipitated asphaltenes in a second solvent with a polarity between a polarity of said first solvent and a polarity of a third solvent capable of dissolving said amount of said asphaltenes completely;
 - determining a proximity to a threshold of instability value of said petroleum residua, wherein said threshold of instability value relates to an amount of said asphaltenes dissolved in said second solvent; and
 - processing said petroleum residua under conditions that maintain a value for said petroleum residua that avoids said threshold of instability value.
95. A method of processing petroleum residua as described in claim 94, wherein said step (d) of determining a proximity to a threshold of instability value of said petroleum residua comprises utilizing a value of K_S for said petroleum residua, wherein K_S is a ratio of a solvated core material size to a core material size for solvated asphaltenes, thus representing an amount of solvent adsorbed around said core material, and wherein said step (e) of processing said petroleum residua comprises processing said petroleum residua under conditions that maintain K_S equal to or greater than about 1.1.
96. The method of processing petroleum residua as described in claim 94, wherein said step (a) of providing an amount of said petroleum residua having solvated asphaltenes dispersed in said petroleum residua comprises selecting said petroleum residua from the group consisting of coal tars, shale oils, tar sand bitumen, asphalts, and heavy oils.
97. The method of processing petroleum residua as described in claim 95, wherein $K_S = (1/(1-\chi_{ey}))$, wherein χ_{ey} is a weight fraction of heptane asphaltenes soluble in cyclohexane.

98. The method of processing petroleum residua as described in claim 94, wherein said step (b) of precipitating an amount of said asphaltenes from said amount of said petroleum residua with a first solvent comprises selecting said first solvent from the group consisting of iso-octane, pentane, hexane, and heptane.
99. The method of processing petroleum residua as described in claim 94, wherein said step (c) of dissolving at least part of said amount of said precipitated asphaltenes in a second solvent comprises selecting said second solvent from the group consisting of cyclohexane, pentane, hexane, heptane, and heptane:toluene (1:1) (v:v).
100. A method of processing petroleum residua as described in claim 94 wherein said step (d) of determining a proximity to a threshold of instability value of said petroleum residua comprises utilizing a value for a ratio of an amount of said asphaltenes dissolved in said second solvent to said amount of precipitated asphaltenes.
101. A liquid distillate produced in accordance with the method of claim 94.
102. A method of analyzing petroleum residua comprising the steps of:
- mixing an amount of said petroleum residua W_a into an amount of aromatic solvent V_s ;
 - titrating aromatic solvent soluble components in said amount of aromatic solvent V_s with an amount of weak aliphatic solvent V_t until a flocculation occurs;
 - calculating a flocculation ratio at infinite dilution FR_{max} from an equation $FR_{max} = V_s / (V_s + V_t)$;
 - calculating a dilution concentration at zero aromatic solvent level C_{min} from an equation $C_{min} = W_a / (V_s + V_t)$ wherein $V_s = 0$;
 - determining a peptizability of asphaltenes p_a from an equation $p_a = 1 - FR_{max}$; and
 - determining a value of p_a / C_{min} .

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103. The method of analyzing petroleum residua as described in claim 102, wherein said step (a) of mixing an amount of said petroleum residua W_a into an amount of aromatic solvent V_s comprises mixing an amount of said petroleum residua into an amount of toluene.
104. The method of analyzing petroleum residua as described in claim 102, wherein said step (b) of titrating aromatic solvent soluble components in said amount of aromatic solvent V_s with an amount of weak aliphatic solvent V_t until a flocculation occurs comprises titrating aromatic solvent soluble components in an amount of aromatic solvent V_s with an amount of iso-octane until a flocculation occurs.
105. The method of analyzing petroleum residua as described in claim 102, further comprising the step of comparing said value of p_a/C_{min} for toluene soluble components with a threshold of instability p_a/C_{min} being within the range of about 0.1 to about 0.4.
106. A method of processing petroleum residua comprising the steps of:
 - a. providing an amount of said petroleum residua W_a having solvated asphaltenes dispersed in said petroleum residua;
 - a. mixing said amount of said petroleum residua W_a into an amount of aromatic solvent V_s ;
 - b. titrating aromatic solvent soluble components in said amount of aromatic solvent V_s with an amount of weak aliphatic solvent V_t until a flocculation occurs;
 - d. determining a proximity to a threshold of instability value of said petroleum residua, as a result of said steps (a) of providing an amount of said petroleum residua W_a having solvated asphaltenes dispersed in said petroleum residua and (b) of mixing said amount of said petroleum residua W_a into an amount of aromatic solvent V_s , utilizing a value of K_F , wherein K_F is a ratio of an average size of a plurality of solvated core materials with an associated solvent to an average size of a plurality of solvated core materials, thus representing an amount of said associated solvent associated with said plurality of solvated core materials; and

- e. processing said petroleum residua under conditions that maintain K_F equal to or greater than about 1.4.
107. The method as described in claim 94 or 106, further comprising processing said petroleum residua under conditions that maintain a solvation shell value of K equal to or greater than about 1.5, wherein $K = K_S \cdot K_F$, wherein K_S is a ratio of a solvated core material size to a core material size for solvated asphaltenes, thus representing an amount of solvent adsorbed around said core material, and K_F is a ratio of an average size of a plurality of solvated core materials with an associated solvent to an average size of a plurality of solvated core materials, thus representing an amount of said associated solvent associated with said plurality of solvated core materials.
108. The method of processing petroleum residua as described in claim 106, wherein $K_F = (1/(1-p_a))$, wherein p_a is a value of peptizability of asphaltenes.
109. A liquid distillate produced in accordance with the method of claim 106.
110. The method as described in claim 94, 102 or 106, further comprising the step of predicting a proximity of said petroleum residua to coke formation.
111. The method as described in claim 94, 102, or 106, further comprising the steps of:
- a. selecting distillation parameters of said petroleum residua to avoid reaching a threshold of instability;
 - b. distilling said petroleum residua using said distillation parameters; and
 - c. avoiding said threshold of instability.
112. The method as described in claim 111, wherein said step (a) of selecting distillation parameters of said petroleum residua to avoid reaching said threshold of instability comprises selecting distillation parameters to avoid coke formation.

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113. The method as described in claim 111, further comprising the step of maintaining continuous distillation of said petroleum residua.
114. The method as described in claim 111, wherein said step (b) of distilling said petroleum residua using said distillation parameters comprises distilling said petroleum residua to closer proximity of said threshold of instability compared to typical distillation parameters.
115. The method as described in claim 94, 102 or 106, further comprising the steps of:
 - a. selecting distillation parameters for said petroleum residua to reach a predetermined level of stability;
 - b. distilling said petroleum residua using said distillation parameters; and
 - c. continuing distillation of said petroleum residua until reaching said predetermined level of stability.
116. The method as described in claim 115, wherein said step (c) of reaching said predetermined level of stability comprises initiating formation of a predetermined amount of coke.
117. The method as described in claim 115, further comprising the step of maintaining continuous distillation until reaching said predetermined level of stability.
118. The method as described in claim 115, further comprising the step of determining a hydrogen-carbon ratio of said petroleum residua.
119. The method as described in claim 115, further comprising the step of determining a total amount of coke, which may form from said petroleum residua.
120. The method as described in claim 115, further comprising the step of increasing output of liquid distillate per unit amount of said petroleum residua.

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121. The method as described in claim 115, further comprising the step of decreasing an amount of energy used per unit of liquid distillate produced.
122. The method as described in claim 115, further comprising the step of reducing an amount of emissions generated per unit of liquid distillate produced.
123. The method as described in claim 122, wherein said step of reducing said amount of emissions generated per unit of liquid distillate produced comprises reducing carbon dioxide emissions.
124. The method as described in claim 115, further comprising the step of increasing a purity of at least a portion of liquid distillates produced.
125. A liquid distillate produced in accordance with the method of claim 102.

The following is an examiner's statement of reasons for allowance:

the closest prior art:

Acevedo (Asphaltenes, 1995), describing "a unified view of the colloidal nature of asphaltenes" and a procedure for precipitating asphaltenes from a 1:1 mixture of toluene and crude oil with heptane and analysis of the precipitated asphaltenes;

Wiehe (Fuel Science & Technology International, 1996), disclosing "the solubility and insolubility of heavy oils and their fractions in dilute mixtures with various solvents ... to characterize heavy oil interactions" with the separation scheme for unconverted and converted residues, including heptane filtration for insoluble asphaltenes;

Yarranton et al. (AIChE Journal, 1996), describing "molar mass distribution and solubility modeling of asphaltenes" with determining dependence of asphaltene-toluene mixture density on asphaltene concentration and determining an average density of asphaltene subfractions vs. mass ratio of insoluble subfraction to total asphaltenes;

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McLean et al. (Journal of Colloid and Interface Science, 1997) disclose “effects of asphaltene aggregation in model heptane-toluene mixtures on stability of water-in-oil emulsions” with determining stability of emulsions via “the amount of water resolved after a 24-h gravitation period” and defining effect of oil aromaticity on asphaltene state of aggregation and resulting interfacial activity;

Laux et al. (Erdoel, Erdgas, Kohle, 1998) describe “boundaries of colloid stability of crude oils” with investigation of strong intermolecular interactions between different organic compounds in asphaltenes: “using the Hansen three-component solubility parameters the stability of an asphaltene dispersion in organic solvents can be described by a spherical solubility region with a radius of about 5 δ -units”;

Schabron et al. (Petroleum Science and Technology, 1998) disclose “the solubility and three-dimensional structure of asphaltenes”, in particular a solubility profile of asphaltene molecules with particular molecular weight and phase diagram for asphaltene molecules;

does not teach or fairly suggest method steps recited in claims 94, 102 and 106 of the instant application.

Another pertinent art is not prior to the instant application:

Castillo et al. (Petroleum Science and Technology, 2001) review “new techniques and methods for the study of aggregation, adsorption, and solubility of asphaltenes” and discuss “impact of these properties on colloidal structure and flocculation”;

Pillon (Petroleum Science and Technology, 2001) discusses “effect of experimental conditions and solvents on the precipitation and composition of asphaltenes”, with emphasis on analysis n-heptane insolubles, effect of pentane on their solubility and toluene soluble asphaltenes.

Therefore, **claims 94-125** are allowed. The new numbering of claims is 1 through 32.

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Any comments considered necessary by applicant must be submitted no later than the payment of the issue fee and, to avoid processing delays, should preferably accompany the issue fee. Such submissions should be clearly labeled "Comments on Statement of Reasons for Allowance."

Any inquiry concerning this communication or earlier communications from the examiner should be directed to Yelena G. Gakh, Ph.D. whose telephone number is (571) 272-1257. The examiner can normally be reached on 9:30 am - 6:00 pm.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Jill A. Warden can be reached on (571) 272-1267. The fax phone number for the organization where this application or proceeding is assigned is 703-872-9306.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see <http://pair-direct.uspto.gov>. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free).

Yelena G. Gakh
3/19/04

Handwritten signature of Yelena G. Gakh in black ink.